

**FORMALDEHYDE-FREE DURABLE PRESS FINISHING AGENT**

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**INVENTOR: SEUNG IL KIM**

**BACKGROUND OF THE INVENTION**

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**Field of the Invention**

[01] The present invention relates to a durable press finishing agent containing no formaldehyde harmful to the human body, and more particularly, to a durable press finish composition, which contains: glyoxal as a crosslinker; ethylene glycol or diethylene glycol as an agent for modifying the crosslinker; aluminum salt, magnesium salt or citric acid as a catalyst for imparting wrinkle-free performance to fabrics; and sodium tetraborate, sodium peroxoborate, sodium chlorite or sodium hydrogen sulfate as an additive for preventing a reduction in strength of fabrics and for increasing the whiteness of fabrics.

### Background of the Related Art

[02] Durable press finishing, which is also called the wash and wear finishing or the DP finishing, is conducted for the purpose of artificially forming crosslinkages at a non-crystalline region of cellulose forming cotton fabrics to increase the elastic limit of the cotton fabrics, thereby giving wrinkling-free performance and shape stability to the cotton fabrics.

[03] A crosslinker which has been most frequently used till now in the industrial durable press finishing of cotton fabrics is dimethyloldihydroxyethyl urea (DMDHDU) which forms ether-type crosslinkages with the hydroxyl group of cellulose. However, this crosslinker generates formaldehyde harmful to the human body, and thus, is currently used in a limited manner. Currently, there are actively progressed studies on polycarboxylic acids, which form ester-type crosslinkages with the hydroxyl group of cellulose and do not generate formaldehyde. Among them, butanetetracarboxylic acid where four adjacent carbon molecules are attached to carboxylic acid is known as having the most excellent effect. However, this butanetetracarboxylic acid is disadvantageous in that it can cause the eutrophication of lakes or rivers upon the durable press finishing of fabrics with it due to a phosphorus component of sodium hypophosphite known as the

most excellent catalyst. Furthermore, it can cause a change in color tone depending on a dye upon dying and also is highly expensive such that it cannot be used in industrial applications. Thus, there is an urgent need for a crosslinker, which does not 5 generate formaldehyde harmful to the human body and allows the finishing costs of fabrics to be reduced.

[04] Meanwhile, although glyoxal is used in the synthesis of DMDHEU, it may also give excellent wrinkle-free performance by itself. Moreover, glyoxal has various advantages as compared to 10 other crosslinkers. First, it has low costs and thus allows the finishing costs of fabrics to be reduced. Also, it is supplied in a highly stable aqueous solution, and thus, considerably easily handled. In addition, it has little or no poisonous character, and thus, can be used as a crosslinker having no 15 harmful effect on the human body. However, when aluminum salt is used as a catalyst in durable press finishing using glyoxal as a crosslinker, fabrics can give excellent wrinkle-free performance but is disadvantageous in that serious reductions in strength and whiteness of fabrics can be caused.

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#### SUMMARY OF THE INVENTION

[05] Accordingly, has been made to solve the above-mentioned problems occurring in the prior art, and an object of

the present invention is to provide a durable press finish composition wherein glyoxal, which is inexpensive and substantially non-poisonous to the human body, is used as a crosslinker, and a catalyst of a kind and amount, that are selected such that the glyoxal can give fabrics wrinkle-free performance which is equal or superior to the prior crosslinkers, is used.

[06] Another object of the present invention is to provide a durable press finish composition, which can minimize reductions in strength and whiteness of fabrics.

[07] To achieve the above objects, the present invention provides a durable press finish composition, which comprises: a crosslinker for giving wrinkle-free performance to fabrics, which consists of glyoxal dissolved in water at a concentration of 3-10% by weight; an agent for modifying the crosslinker, which is selected from ethylene glycol and diethylene glycol and contained at a molar ratio of 1:1 relative to the glyoxal; a catalyst selected from aluminum sulfate and a mixture of magnesium chloride and citric acid, the aluminum sulfate being contained at a molar ratio of 0.01-0.08 relative to the glyoxal, and the mixture of magnesium chloride and citric acid being contained at a molar ratio of 0.1-0.6 relative to the glyoxal, the citric acid being contained at a molar ratio of 0.1-0.5 relative to the

magnesium chloride; and an additive selected from sodium tetraborate, sodium peroxoborate, sodium chlorite and sodium hydrogen sulfate wherein the sodium tetraborate or sodium peroxoborate is contained at a weight ratio of 0.1-0.6% relative  
5 to the composition, the sodium chlorite is contained at a weight ratio of 0.01-0.06% relative to the composition, and the sodium hydrogen sulfate is contained at a weight ratio of 0.01-0.08% relative to the composition.

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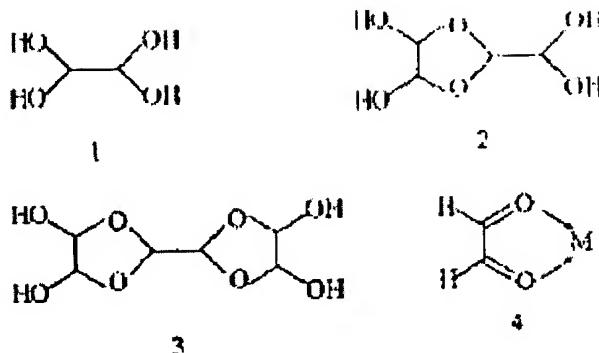
#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[08] Hereinafter, the present invention will be described in detail.

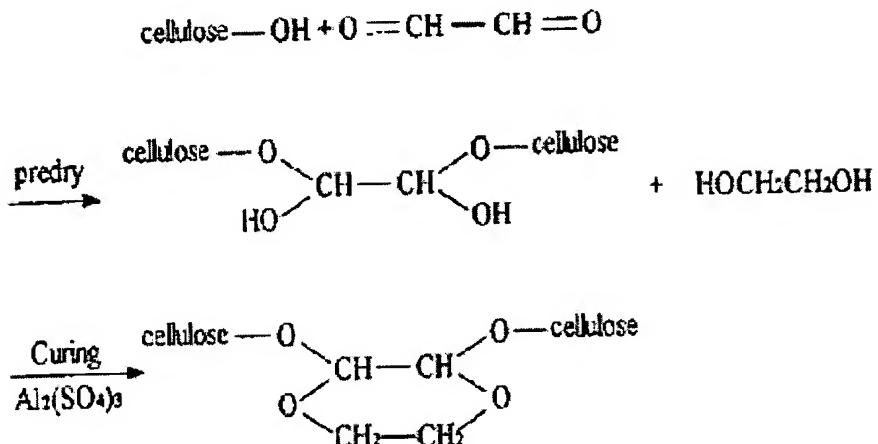
[09] The present invention relates to a durable press finish composition, which contains: glyoxal as a crosslinker for  
15 imparting wrinkle-free performance to fabrics; ethylene glycol or diethylene glycol as an agent for modifying the crosslinker; aluminum salt, magnesium salt or citric acid as a catalyst; and sodium tetraborate, sodium peroxoborate, sodium chlorite or sodium hydrogen sulfate as an additive.

20 [10] Formula 1 below shows the chelation of three compositions of glyoxal available as a 40% aqueous solution with a metal catalyst. Reaction Scheme 1 below shows the reaction between cellulose, glyoxal and ethylene glycol.

[11] [Formula 1]



[12] [Reaction Scheme 1]



5 [13] Formula 2 below shows the structure of ethylene glycol,  
which is used as an agent for modifying the crosslinker in the  
present invention.

[14] [Formula 2]

[15] HOCH<sub>2</sub>CH<sub>2</sub>OH

10 [16] Formula 3 below shows the structure of diethylene  
glycol, which is used as an agent for modifying the crosslinker  
in the present invention.

[17] [Formula 3]

[18] HOCH<sub>2</sub>CH<sub>2</sub>OCH<sub>2</sub>CH<sub>2</sub>OH

[19] Formula 4 below shows the structure of aluminum sulfate, which is used as a catalyst in the present invention.

5 As the catalyst, there can be all hydrates where x is 14-18, and preferably 16.

[20] [Formula 4]

[21] Al<sub>2</sub> (SO<sub>4</sub>)<sub>3</sub> · xH<sub>2</sub>O wherein x = 14 to 18.

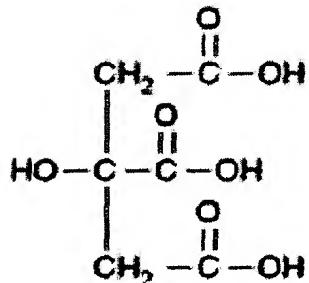
10 Formula 5 below shows the structure of magnesium chloride, which is used as a catalyst in the present invention.

[23] [Formula 5]

[24] MgCl<sub>2</sub> · 6H<sub>2</sub>O

15 [25] Formula 6 below shows the structure of citric acid, which is used as a catalyst in the present invention.

[26] [Formula 6]



[27] Formula 7 below shows the structure of sodium tetraborate, which is used as an additive in the present invention.

[28] [Formula 7]

[29]  $\text{Na}_2\text{B}_4\text{O}_7$

[30] Formula 8 below shows the structure of sodium peroxoborate, which is used as an additive in the present  
5 invention.

[31] [Formula 8]

[32]  $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$

[33] Formula 9 below shows the structure of sodium chlorite, which is used as an additive in the present invention.

10 [34] [Formula 9]

[35]  $\text{NaClO}_2$

[36] Formula 10 below shows the structure of sodium hydrogen sulfate, which is used as an additive in the present invention.

15 [37] [Formula 10]

[38]  $\text{NaHSO}_4$

[39] Formula 11 below shows the structure of sodium phosphate monobasic, which is used as a buffer in the present invention.

20 [40] [Formula 11]

[41]  $\text{NaH}_2\text{PO}_4$

[42] In preparing the durable press finish composition according to the present invention, glyoxal of Formula 1 is

dissolved in water at a weight ratio of 3-10%, to which one of ethylene glycol and diethylene glycol represented by Formulas 2 and 3, respectively, is added at a molar ratio of 1:1 relative to the glyoxal. To this mixture, the catalyst of Formula 4, 5 or 6  
5 is added at a specified amount, and then, the additive of Formula 7, 8, 9 or 10 is added at a specified amount, thereby giving the durable press finish solution composition.

[43] Fabrics are dipped in the prepared solution to sufficiently wet them with the solution, and then, passed through  
10 padding rollers and these procedures are repeated one time. The fabrics wet with the solution are pre-dried on a tenter at 80-100 °C, and then, thermally treated at a temperature of 130-180 °C to form crosslinkages between cellulose molecules in a non-crystalline region of celluloses. The thermally treated fabrics  
15 are sufficiently washed with water at 50 °C, and then, dried on a tenter at a temperature of 80-100 °C, thereby completing the durable press finishing of the fabrics.

[44] In the present invention, glyoxal as a crosslinker for imparting wrinkle-free performance to fabrics is preferably dissolved in water at a concentration of 3-10% by weight. If its  
20 concentration in water is lower than 3% by weight, it cannot impart wrinkle-free performance to fabrics, and if its

concentration in water is higher than 10% by weight, a reduction in strength of fabrics will be caused.

[45] Meanwhile, ethylene glycol or diethylene glycol as an agent for modifying the crosslinker is preferably used at a molar 5 ratio of 1:1 relative to the glyoxal. In this case, ethylene glycol or diethylene glycol causes a change in length and shape of the crosslinkage formed between fibers, and thus, has a positive effect on the wrinkle-free performance of fabrics.

[46] Although all metal salt catalysts or organic acid 10 catalysts serving as Lewis acid in aqueous solution may be used as a catalyst in the present invention, it is preferable to use aluminum sulfate, magnesium chloride, citric acid or a mixture thereof. If aluminum sulfate is used at a molar ratio higher than 0.08 relative to glyoxal as a crosslinker, it will cause a 15 serious reduction in strength and whiteness of fabrics, and if it is used at a molar ratio lower than 0.01 relative to glyoxal, it cannot give fabrics wrinkle-free performance. Also, the amount of aluminum sulfate used will vary depending on the kind of fabrics used. Magnesium chloride, when used alone, cannot give 20 fabrics wrinkle-free performance, but the use of a mixture of magnesium sulfate and citric acid can impart high-level wrinkle-free performance to fabrics. In this case, magnesium chloride is preferably used at a molar ratio of 0.1-0.6 relative to glyoxal,

and citric acid is used at a molar ratio of 0.1-0.5 relative to magnesium chloride.

[47] Furthermore, the additive used in the present invention acts to prevent the reduction of strength and whiteness of treated fabrics by competition with the catalyst in the reaction between cellulose and the catalyst. In the case where aluminum sulfate or a mixture of magnesium chloride and citric acid is used as a catalyst, the additive of Formula 8 or the additive of Formula 9 in the present invention is preferably used at a weight ratio of 0.1-0.6% relative to the solution composition. If the additive is used at the amount of less than 0.1% by weight, it cannot serve to prevent the reduction of strength and whiteness of fabrics, and if it is used at the amount of more than 0.6, it will adversely affect the wrinkle-free performance of fabrics. In the case where aluminum sulfate or a mixture of magnesium sulfate and citric acid is used as a catalyst, the additive of Formula 10 is preferably used at the amount of 0.01-0.08% by weight relative to the solution composition. In order to give optimal wrinkle-free performance to fabrics while preventing the strength and whiteness of fabrics from being reduced, it is preferable to use a very small amount of sodium phosphate monobasic, a buffer of Formula 12.

[48] As described above, the present invention provides the durable press finish composition wherein glyoxal, which contains no poisonous formaldehyde, has low costs and can give fabrics excellent wrinkle-free performance, is used as a crosslinker, and 5 at the same time, the catalyst and additive of suitably selected kinds and amounts, that are selected such that the crosslinker can give optimal wrinkle-free performance to fabrics, are used.

[49] The present invention will hereinafter be described in further detail by examples and comparative examples. It should 10 however be borne in mind that the present invention is not limited to or by the examples.

[50] Example 1

[51] Gloxal as a crosslinker was dissolved in water at a concentration of 5% by weight, to which aluminum sulfate as a 15 catalyst was added at a molar ratio of 0.04 relative to the glyoxal, thereby producing the finishing solution 1. To the finishing solution 1, each of ethylene glycol and diethylene glycol was added at a molar ratio of 1:1 relative to the glyoxal, thereby producing the finishing solutions 2 and 3, respectively. 20 Then, 100% cotton fabrics are finished with each of the finishing solutions 1, 2 and 3 by a pad-dry-cure method. In this case, the pad was carried out in a 2dip-2nip manner such that a wet pick-up

is set at about 95%. The dry is carried out at 85 °C for 3 minutes and the cure at 150°C for 3 minutes.

[52] The fabrics which had been finished under the above conditions were measured for their wrinkle recovery angle 5 according to AATCC Test Method 66-1998. Also, their tensile strength was measured according to ASTM D 1682-64 and their whiteness index was measured by X-Rite spectrophotometer according to ASTM E 313. The amount of free formaldehyde from the finished fabrics was measured according to JIS L 1041 A.A 10 METHOD B. The results showed that the wrinkle recovery angle of the finished fabrics was superior to that of fabrics finished with the prior durable press finishes, and their tensile strength and whiteness index were similar to those of the fabrics finished with the prior durable press finishes. However, there was no 15 detection of formaldehyde from the finished fabrics.

[53] Example 2

[54] The additive of Formula 10 was added to the finishing solution 2 produced in Example 1, at a molar ratio of 0.25-1.5 relative to aluminum sulfate as a catalyst, thereby producing a 20 finishing solution. 100% cotton fabrics were finished with this finishing solution and measured for their physical properties in the same manner as in Example 1. The results showed that the wrinkle recovery angle of the finished fabrics was gradually

reduced with an increase in concentration of the additive, but higher than that of fabrics finished with the prior durable press finishes. Also, it could be found that their tensile strength and whiteness index were gradually increased with an increase in  
5 concentration of the additive. Thus, it could be found that all the physical properties of the fabrics finished with this finishing solution were equal or superior to fabrics finished with the prior durable press finishes. As in Example 1, there was no detection of formaldehyde in the fabrics finished with the  
10 solution of Example 2.

**[55] Example 3**

**[56]** Glyoxal as a crosslinker was dissolved in water at a concentration of 5%, to which a mixture of magnesium chloride and citric acid as a catalyst was then added, thereby producing a  
15 finishing solution. 100% cotton fabrics were finished with this finishing solution in the same manner as in Example 2. In this case, the molar ratio of magnesium chloride to glyoxal was 0.2, and the molar ratio of citric acid to magnesium chloride varied in the range of 0.1-0.5.

20 **[57]** The finished cotton fabrics were measured for their physical properties in the same manner as in Example 1. The results showed that the wrinkle recovery angle of the fabrics finished with the solution of Example 3 was superior to fabrics

finished with the prior durable press finishes. But their tensile strength and whiteness index were somewhat reduced, and there was no detection of formaldehyde in the fabrics finished with the solution of Example 3.

5        [58]    Example 4

[59] To the finishing solution of Example 3 where the molar ratio of citric acid to magnesium chloride was set at 0.2, each of additives of Formulas 7 and 8 was added at amounts varying in the range of 0.1-0.6% by weight relative to the weight of the 10 finishing solution, thereby producing a finishing solution. 100% cotton fabrics were finished with this finishing solution, and measured for their physical properties. The results showed that their wrinkle recovery angle was somewhat lower than that of Example 3, but higher than that of fabrics finished with the 15 prior durable press finishes. Also, their tensile strength and whiteness index were equal to those of fabrics finished with the prior durable press finishes. Furthermore, there was no detection of formaldehyde in the fabrics finished with the solution of Example 3.

20        [60]    Example 5

[61] Glyoxal as a catalyst was dissolved in water at a concentration of 5%, to which aluminum sulfate as a catalyst was added at a molar ratio of 0.04 relative to the glyoxal. To this

solution, each of additives of Formulas 7 and 8 was added at amounts varying in the range of 0.1-0.6% by weight relative to the weight of the solution, thereby producing a finishing solution. 100% cotton fabrics were finished with the finishing 5 solution and measured for their physical properties in the same manner as in Example 1. The results showed that the wrinkle recovery angle of the finished fabrics was slightly reduced with an increase in concentration of the additives, but higher than that of fabrics finished with the prior durable press finishes.

10 As in Example 1, there was no detection of formaldehyde in the fabrics finished with the solution of Example 5.

**[62] Example 6**

**[63]** Glyoxal as a crosslinker was dissolved in water at the concentration of 5%, to which each of a mixture of magnesium 15 chloride and citric acid, and aluminum sulfate, as a catalyst, was added. To this solution, sodium chlorite of Formula 9 as an additive was added at amounts varying in the range of 0.01-0.06% by weight relative to the weight of the solution, thereby producing finishing solutions. 100% cotton fabrics were finished 20 with each finishing solution in the same manner as in Example 1. Also, a buffer of Formula 11 was added to the respective finishing solutions at a small amount. 100% cotton fabrics were finished with the finishing solutions containing the buffer in

the same manner, and compared to the fabrics finished with the finishing solution containing no buffer. The fabrics finished with such finishing solutions were measured for their physical properties in the same manner as in Example 1. The results 5 showed that all the physical properties (particularly, whiteness index) of the fabrics finished with the solutions of Example 5 were superior to fabrics finished with the prior durable press finishes. Also, there was no detection of formaldehyde in the fabrics finished with the solutions of Example 5.

10 [64] As described above, the present invention provides the durable press finish composition wherein glyoxal, which contains no formaldehyde harmful to the human body, has low costs and can give excellent wrinkle-free performance to fabrics, is used as a crosslinker, and the catalyst and additive of kinds and amounts 15 suitable to give optimal wrinkle-free performance to fabrics are used. Thus, according to the durable press finish composition of the present invention, serious reductions in strength and whiteness of fabrics, which are problems occurring in the prior durable press finishes, can be prevented. In addition, the 20 inventive durable press finish composition can give fabrics wrinkle-free performance that is equal or superior to the prior crosslinkers.

[65] While the present invention has been described with reference to the particular illustrative embodiments, it is not to be restricted by the embodiments but only by the appended claims. It is to be appreciated that those skilled in the art can change or modify the embodiments without departing from the scope and spirit of the present invention.